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Study of electrical properties of Ni-phyllosilicate nanoscrolls with reduced Ni nanoparticles

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Catalyst materials consisting of metal (Fe, Co, Ni) nanoparticles placed on an oxide carrier possess great interest for application in a wide range of chemical process [1]. These structures can be strong electric fields sources – a single-isolated metal ball with a radius of 1 nm with a charge of 1 electron creates a field of about 10^9 V/m. Such high fields might have significant influence on chemical reaction potential barrier. Hence, the problem relies in the synthesis of stable catalytically active metallic nanoparticles arrays. One way to create such arrays is laser electro-dispersion (LED) [2]. In our work, another method to form isolated metal nanoparticles was implemented: they were reduced out of the Ni-doped phyllosilicate nanoscrolls in hydrogen flow.

The $(Ni_xMg_{1-x})_3Si_2O_5(OH)_4$ (x = 0.67 and 1) nanoscrolls were prepared by hydrothermal treatment (350 ºC, 20 MPa, 12 h, 0.25 M NaOH water solution) of coprecipitated of Mg and Ni hydroxides in the presence of amorphous SiO_2. Further, the samples were annealed in a reducing medium (hydrogen) in the 400-900 ºC temperature range, which made it possible to obtain metal nanoparticles incorporated into a silicate matrix. The samples morphology was studied by the transmission electron microscopy (TEM), Figure 1.

To inject the charge into the sample and to study the charge leakage, the Kelvin probe force microscopy (KPFM) method was used. It was found that, initially, the nanoscroll potential is about 1 mV, and the area around it has a potential of 3 mV (Fig. 2b). After charging by voltage pulse application, the nanoscroll potential increased to 3 mV, and the surrounding area – to 9 mV (Fig. 2c). With time, the potentials slowly decreased to 2 mV and 7 mV, respectively (Fig. 2d, red line).

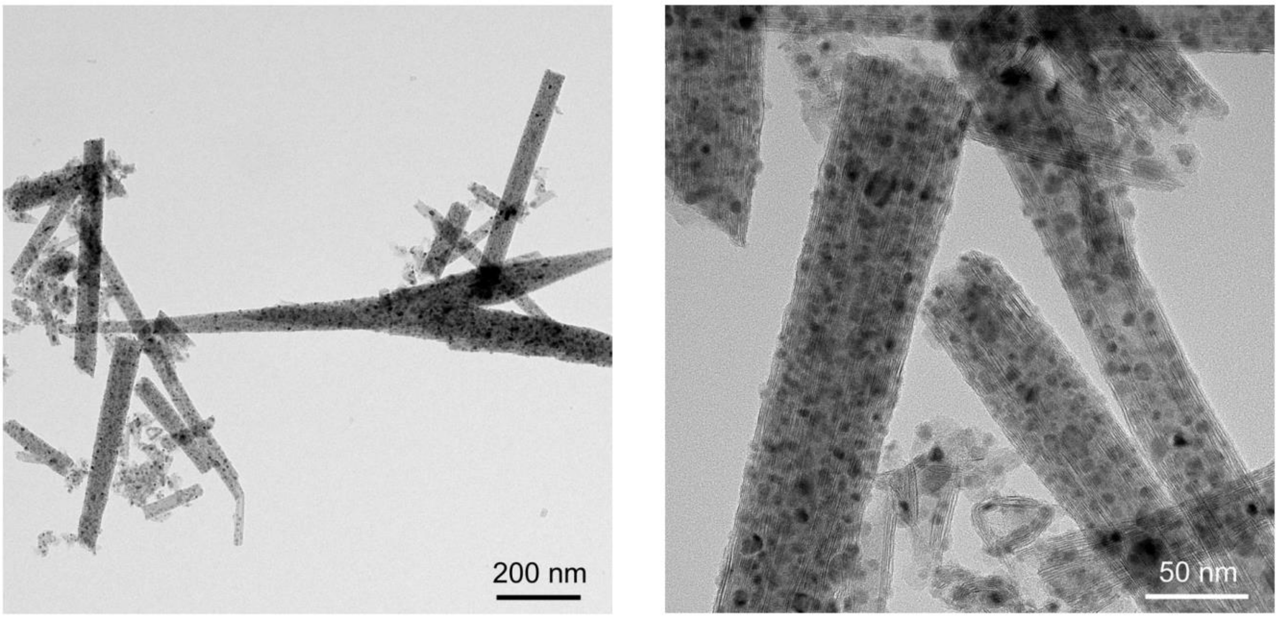
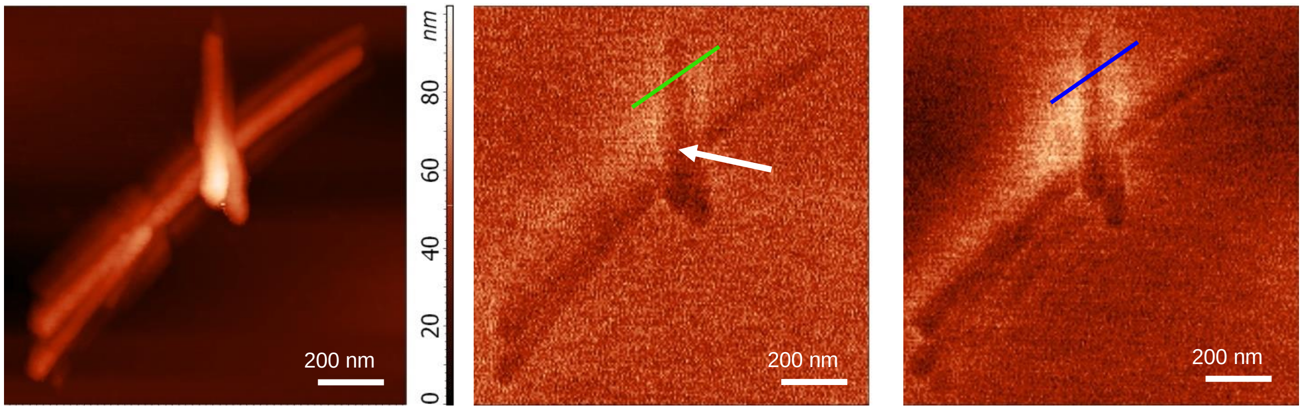
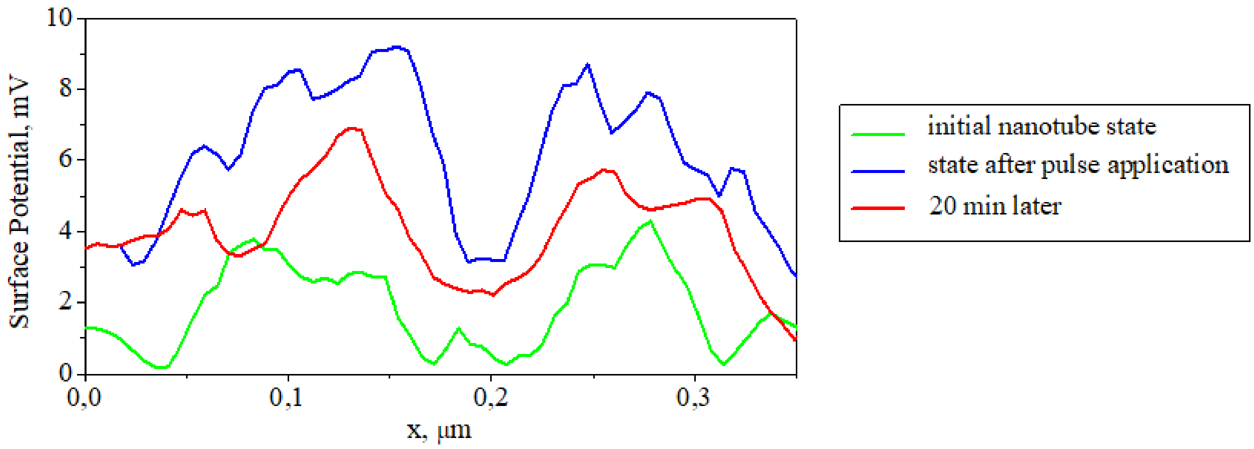


Figure 1. TEM micrographs of $Ni_2MgSi_2O_5(OH)_4$ nanoscrolls after partial reduction of Ni.



(a) (b) (c)



(d)

Figure 2. KPFM topography image (a) and surface potential map of the Ni(30 nm)/Si substrate with $Ni_3Si_2O_5(OH)_4$ nanoscrolls before (b) and after (c) rectangular voltage pulse application (τ = 30 s, U = +10 V). The surface potential profiles (d) across the uncharged nanoscroll (green), immediately after charge injection (blue) and 20 min later (red). Parameters of visualization: HA_C/W2C+ cantilever, resonant frequency of 17.8 kHz, free / set.point oscillation amplitude of about 8 nm / 5 nm; probe-sample distance at KPFM mode is about 10 nm. Arrows indicate the contact point the charge was injected.

The report will also consider the results of the charge injection experiments (using KPFM and spreading resistance microscopy) for the samples of different Ni, Mg contents as well as annealing temperatures.

1. Q .Yang, Q. Xu, H.L. Jiang, *Chemical Society Reviews*, **118**, 10, 4981 (2017).
2. E.V. Golubina, E.S. Lokteva, K.I. Maslakov, T.N. Rostovshchikova, M.I. Shilina, S.A. Gurevich, V.M. Kozhevin, D.A. Yavsin, *Nanotechnologies in Russia*, **12**, 19 (2017).